Effect of Steam Explosion (SE) Pretreatment on the Contamination of Woody Biomass with Metallic Inhibitors

Jan Szadkowski *, Anna Gałązka, Witold Jan Wardal *

Posted Date: 24 July 2025

doi: 10.20944/preprints202507.2074.v1

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 doi:10.20944/preprints202507.2074.v1

Article

Effect of Steam Explosion (SE) Pretreatment on the Contamination of Woody Biomass with Metallic Inhibitors

Jan Szadkowski [1,*], Anna Gałązka [2] and Witold Jan Wardal [3,*]

[1] Department of Wood Science and Wood Preservation; Institute of Wood Sciences and Furniture, Warsaw University of Life Sciences, 166 Nowoursynowska St., 02-787 Warsaw, Poland

[2] Faculty of Wood Technology, Warsaw University of Life Sciences – SGGW, 166 Nowoursynowska St., 02-787 Warsaw, Poland

[3] Department of Technology and Entrepreneurship in Wood Industry, Institute of Wood Sciences and Furniture, Warsaw University of Life Sciences, 166 Nowoursynowska St., 02-787 Warsaw, Poland

* Correspondence: jan_szadkowski@sggw.edu.pl (J.S.); witold_wardal@sggw.edu.pl (W.J.W.)

Abstract

The aim of the study was to develop an optimum method for investigating the change in the content of selected metals in biomass before and after pretreatment by steam explosion. The study included testing the metal content using an X-ray fluorescence spectrometer (XRF) of metals such as chromium, manganese, iron, nickel, copper and zinc. These metals are considered inhibitors of biological processes occurring during biofuel production such as enzymatic hydrolysis, alcoholic fermentation. In this study, a steam explosion process was carried out on poplar wood biomass at selected temperatures in the range 140°C -205°C. The study found the greatest increase in metal content for materials after SE at 175°C. The significant increase in the accuracy of the determination of metals in the material is influenced by the transfer of the raw material to the liquid state.

Keywords: XRF; poplar; steam explosion; inhibitors; wood

1. Introduction

The development of a zero-emission economy brings with it the need to develop technology that is efficient and as economically viable as possible, while ensuring that the environmental impact of the technology developed is minimal. One promising energy generation technology is the conversion of lignocellulosic biomass to liquid biofuels such as bioethanol [1]. Another method of obtaining energy feedstock in low-carbon biofuel technology is to gasify biomass and then use the resulting gas mixture for power generation or chemical synthesis [2,3].

Conversion can be carried out on any lignocellulosic biomass. Its yield will depend on its chemical composition, moisture content, and pretreatment process. The lignocellulosic material is one whose main cell wall building block is a lignocellulosic complex consisting of three basic substances: cellulose, hemicelluloses and lignin [4].

Depending on the raw materials used and the technology, biofuels are subdivided into three generations (there are three generations of biofuels where generation I fuels use lignocellulosic material such as wood or cereal grains for energy production (combustion) or to produce energy raw materials (ethanol), generation II are lignocellulosic materials that do not compete with the food industry or are production waste from other industries with no further use (straw, post-consumer wood). Generation III, which is referred to as the biomass of the future, is based on raw materials that are not used in other industries, are characterised by high growth and low environmental impact and include fuels derived from algae and trees genetically modified for rapid growth [5–8].

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Efficient extraction of energy raw materials from lignocellulosic biomass requires the use of pretreatment, i.e. a process to increase the availability of chemicals for subsequent processes. Mechanical, chemical, physico-chemical and biological pretreatments are distinguished [9–12].

During the pretreatment and conversion of lignocellulosic materials, organic and inorganic chemical compounds are formed that can hinder the conversion of biomass in subsequent stages. They are considered to be inhibitors of biological processes, which are part of the basis of environmentally friendly technologies. Such compounds include, for example, furfural, hydroxymethylfurfural, organic acids (lactic acid, oleic acid, levulinic acid) mineral acids, e.g. carbonic acid, metal ions such as nickel, copper, iron, magnesium and chromium. These compounds impair the action of enzymes, enzyme analogues and the organisms themselves used in biomass conversion, e.g. yeast used for ethanol production [9,12–15].

Steam explosion (SE) is considered the most promising and promising pretreatment for lignocellulosic biomass biofuel technology. The steam explosion method uses the action of saturated steam, water or ammonia and carbon dioxide at elevated temperature and pressure. The best-studied method to date is steam action. The steam explosion is carried out in a special high-pressure autoclave. The test material, sealed in the autoclave tank, is subjected to high temperature and high pressure. This is followed by rapid decompression to atmospheric pressure [16]. As a result of SE, lignin is degraded and cellulose and part of the hemicelluloses are broken down to simple sugar such as xylose, galactose, mannose, glucose. Material prepared in this way is more accessible to hydrolytic enzymes [17].

The steam explosion method is considered one of the most effective and efficient pretreatment methods. The process requires 70% less energy than physical methods [18] Above that, it allows for a reduction in waste and recycling costs [19]. However, some of the haemicelluloses are degraded as a result of high temperatures. This is a limitation due to the loss of monosaccharides that would have been formed from their degradation. The effects of high temperature and pressure not only leach the mineral compounds contained in the wood, but also oxidise the metal from the apparatus and cause corrosion. The released metal compounds penetrate into the test material and, along with the decomposition products of the biomass components, constitute metal inhibitors that can hinder and slow down the enzymatic hydrolysis process. The amount of inhibitors that entered the wood material depends on the conditions under which the steam explosion process was carried out. During the steam explosion experiment, care must be taken to select the appropriate process parameters. The temperature at which the steam explosion is carried out ranges from 160°C to 290°C. The pressure ranges from 5 to 35 bar, depending on the temperature and apparatus [20].

The aim of this study was to analyse the content of heavy metals in the wood of 5-year-old genetically modified poplar pretreated by steam explosion at selected temperatures. In order to realise the aim of the study, the contents of selected heavy metals were compared in ashed wood material pretreated by steam explosion at temperatures of 160-205°C. Additional verification was provided by measurements made on ash samples dissolved in nitric acid.

2. Materials and Methods

2.1. Materials

Wood of fast-growing poplar five years after planting on the experimental site was used for the study. The harvested material was dried in a laboratory dryer to an absolute moisture content of 0%. After drying, the material was debarked, selected to remove defects such as knots, false heartwood, etc. The material thus selected was ground using a laboratory knife mill. The poplar chips thus obtained were separated into fractions using laboratory sieves. The 0.43-1.0 mm fraction was used for further analyses and experiments.

2.2. Steam Explosion (SE)

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The steam explosion was performed on 20 g of 0.43-1.0 mm chips flooded with distilled water so that there was at least 10 ml of water per 1 g of dry chips. In order to remove air from the wood pores, the wood shavings were pre-poured in a beaker with 1/3 of the volume of water and heated at 90 °C with stirring for 20 min. The material thus prepared was placed in a stainless steel high-pressure laboratory autoclave fitted with a 5 cm diameter spherical valve and a receiver in which a vacuum is set before sudden decompression of the autoclave. The autoclave and the valve are suitable for operation up to 250 °C. The autoclave itself has a built-in electric heating system. The experiment was conducted at four temperatures: 160, 175, 190 and 205 °C. After the steam explosion, the material was transferred to a receiver, from where it was carefully rinsed with distilled water into a glass beaker (2000 cm^3). Using a tissue filter placed on a Büchner funnel connected to a mechanical vacuum pump, the solid fraction was separated from the liquid fraction. Three experiments were carried out for each temperature variant.

2.3. Measurement of Heavy Metal Content

After steam explosion treatment, the material was ashed in a muffle furnace at 600°C prior to metal content analysis. The heavy metal content was measured by X-ray fluorescence XRF. For this purpose, a fluorescence spectrometer from Spectro Mixed M was used. Technical data of the XRF X-ray fluorescence spectrophotometer from Spectro Mixed M (data was provided by the manufacturer in the user manual).

Excitation system:

- maximum power: 30 W
- maximum current: 0.8 mA
- maximum voltage: 50 kV
- X-ray tube with air-cooled molybdenum anode

The measurement chamber has a measurement table positioning accuracy of 2.5 μm. The chamber dimensions are 540 x 600 x 2500 mm. The image on the computer monitor is displayed using a dual camera system. The ashes obtained from the combustion were placed on a clean and appropriately labelled sheet of paper. The material was arranged so that the tops of the samples were on one level. The sheet with the ashes was transferred to the test table of the XRF spectrometer and an initial measurement was made.

Three measurement points were determined on each sample (using a 2x2mm aperture. Each point was exposed for 300 seconds. The relative changes in the content of heavy metals such as chromium, iron, nickel, copper, manganese and zinc expressed as percentages were compared. In order to accurately verify the changes in the metal content of the steam-blasted material, an analysis of the metal content of ash dissolved in nitric acid was carried out. Such an assay allows measurement of the content of selected metals with greater accuracy due to the ability to accurately calibrate measurements of liquid samples. Samples of approximately 0.1 g of ash were weighed out on a laboratory balance. Volume 1 cm^3 of concentrated nitric acid was added to each sample and boiled until dissolved. The ash dissolved after about 4 hours of boiling. The solution thus obtained was then cooled and neutralised by adding 2 cm^3 of ammonia water (25%). Three drops (0.3 cm^3) of the solution were tested from each sample. The drops were placed with a pipette on a dish to be placed on the measuring table of the spectrometer. One measuring point was determined on each drop. Each sample was exposed for 300 seconds. The process was repeated three times.

3. Results

This section may be divided by subheadings. It should provide a concise and precise description of the experimental results, their interpretation, as well as the experimental conclusions that can be drawn.

3.1. Weight Loss

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During the experiments, mass loss due to steam explosion was determined. The results are presented in Table 1.

Table 1. Metal content in wood measured in ash.

Steam Explosion Temperature [°C]	Average weight of dry samples before SE [g]	Mass of wet chips after steam explosion [g]	Dry shavings weight after draining [g]	Material weight loss [g]	Material weight loss [%]
160	18.988	42.82	17.984	1.003	5.3
175	18.892	27.83	16.976	1.915	10.1
190	18.926	32.23	16.760	2.166	11.4
205	18.894	22.3	16.725	2.169	11.5

The mass loss indication shows that the greatest mass loss is for the material pre-treated at 205 °C.

3.2. Metal Content After Steam Explosion

Tables 2 and 3 present the results of the content of selected metals in the obtained ash and per *g* of ash and *g* of wood chips.

Table 2. Metal content in wood measured in ash.

Temperature [°C]	Sample	Metal content in wood x10^3 [g/g]					
		Cr	Fe	Ni	Cu	Mn	Zn
160	1	0.21354	1.33675	0.24320	0.18370	0.21727	4.30166
	2	0.09325	0.60893	0.11964	0.09656	0.15433	1.34980
	3	0.12496	1.27008	0.25360	0.20179	0.22007	2.87445
	Average	0.14392	1.07192	0.20548	0.16069	0.19723	2.84197
	Standard deviation (SD)	0.06235	0.40234	0.07452	0.05627	0.03717	1.47619
175	1	0.07784	3.22806	6.78434	0.21676	0.16611	6.78434
	2	0.17013	4.07924	5.30812	0.03362	0.13663	5.30812
	3	0.18911	10.19221	7.30651	0.03202	0.16961	7.30651
	Average	0.14569	5.83317	6.46632	0.09413	0.15745	6.46632
	SD	0.05953	3.79896	1.03646	0.10620	0.01812	1.03646
190	1	0.21657	0.40782	3.09069	0.21657	0.40782	3.09069
	2	0.16748	0.31904	2.73041	0.16748	0.31904	2.73041
	3	0.30237	0.44505	4.70763	0.30237	0.44505	4.70763
	Average	0.22880	0.39063	3.50958	0.22880	0.39063	3.50958
	SD	0.06827	0.06474	1.05307	0.06827	0.06474	1.05307
205	1	0.09855	0.08932	3.05353	0.09855	0.08932	3.05353
	2	0.12725	0.10891	3.76234	0.12725	0.10891	3.76234
	3	0.19807	0.14038	6.26534	0.19807	0.14038	6.26534
	Average	0.14129	0.11287	4.36040	0.14129	0.11287	4.36040
	SD	0.05123	0.02576	1.68736	0.05123	0.02576	1.68736
Nativ	1	0.21682	2.93306	1.46376	0.21682	2.93306	1.46376
	2	0,10206	1.27106	0.44431	0.10206	1.27106	0.44431
	3	0.10926	1.34340	0.56606	0.10926	1.34340	0.56606
	Average	0.14271	1.84917	0.82471	0,14271	1.84917	0.82471
	SD	0.06428	0.93937	0.55677	0.06428	0.93937	0.55677

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The results of the metal content determinations in ash indicate that the content of the analyzed elements varies ambiguously within the analyzed material. We cannot conclude that the content of these substances is highest at a specific temperature.

Table 3. Metal content in wood measured after dissolving the ash in acid.

Temperature [°C]	Sample	Metal content in wood x10^3 [g/g]					
		Cr	Fe	Ni	Cu	Mn	Zn
160	1	0.00339	0.03274	0.01234	0.00993	0.00619	0.15259
	2	0.00384	0.03343	0.01294	0.01029	0.00642	0.15530
	3	0.00384	0.03343	0.01294	0.01029	0.00642	0.15530
	Average	0.00369	0.03320	0.01274	0.01017	0.00634	0.15440
	SD	0.00026	0.00039	0.00034	0.00021	0.00014	0.00156
175	1	0.00525	0.22814	0.04173	0.02887	0.00954	0.87618
	2	0.00786	0.23130	0.04652	0.03362	0.01345	0.88627
	3	0.00714	0.22566	0.04391	0.03202	0.01160	0.87912
	Average	0.00675	0.22837	0.04405	0.03150	0.01153	0.88052
	SD	0.00135	0.00282	0.00240	0.00242	0.00196	0.00519
190	1	0.01595	0.11436	0.02844	0.02864	0.02717	0.23926
	2	0.01044	0.11934	0.02400	0.02610	0.02054	0.33986
	3	0.00903	0.11655	0.02137	0.02371	0.01766	0.34630
	Average	0.01181	0.11675	0.02461	0.02615	0.02179	0.30847
	SD	0.00366	0.00249	0.00358	0.00246	0.00488	0.06003
205	1	0.00167	0.02252	0.00445	0.00595	0.00241	0.19810
	2	0.00167	0.02260	0.00420	0.00577	0.00192	0.20345
	3	0.00234	0.02479	0.00585	0.00732	0.00328	0.20696
	Average	0.00189	0.02330	0.00483	0.00635	0.00254	0.20284
	SD	0.00039	0.00129	0.00089	0.00085	0.00069	0.00446
Nativ	1	0.01462	0.02052	0.02519	0.02377	0.03020	0.03582
	2	0.01341	0.01815	0.02173	0.02018	0.02600	0.02993
	3	0.00934	0.01544	0.01693	0.01422	0.02268	0.02512
	Average	0.01246	0.01803	0.02128	0.01939	0.02629	0.03029
	SD	0.00277	0.00254	0.00415	0.00482	0.00377	0.00536

The metal content determined after dissolving the ash in nitric acid showed different values than those found in the ash itself. The trend in the content of individual metals in the material is the same for both assay variants.

The results of the metal content in the material after steam explosion treatment indicate ambiguous behavior of the tested elements. The highest content of metal inhibitors is observed at 175°C. At this temperature, iron, nickel, zinc, and copper exhibit the highest content. The content of potential metallic inhibitors in the material after the steam explosion at 200°C is the lowest except for zinc where the value is higher than in the native material and after the process at 160°C. In the case of chromium and manganese, native wood shows the highest levels of these elements. Chromium is most abundant outside the native material in the wood after the SE process at 190°C.

4. Discussion

4.1. Weight Loss

During pretreatment with a steam explosion, the mass of the material on which the process takes place is reduced. In the study presented here, a weight loss of up to 11.5 % was achieved. This value depends on the process temperature and the biomass being treate [21,22]. The value of this process is most effective at temperatures above 240°C [21,23].

 doi:10.20944/preprints202507.2074.v1

This is a result of the thermal strength of the biomass components. The least resistant structural components are hemicelluloses, with amorphous lignin decomposing at higher temperatures, followed by cellulose, which can withstand 400°C [24].

The results obtained are consistent with the literature data.

4.2. Metal Content After Steam Explosion

Zielenkiewicz et al. [25] pointed out in their work that the key to determining accurate metal content in woody biomass using XRF is to calibrate the method to the specific wood species being analysed. The method of analysing samples after ashing is more accurate than analysing unashed wood, but is not free of drawbacks. The most accurate results are obtained for ash dissolved in nitric acid. The results obtained during the realisation of our work show a high variability in ashed versus ashed and dissolved material in nitric acid. The problem of accurately determining the metal content of non-metallic samples can arise from the heterogeneous structure of the material being analysed [26–29].

Krutul et al. [30] indicate that the heavy metal content of poplar biomass due to pretreatment processes such as steam explosion (SE) and liquid hot water (LHW) increases with the temperature of the treatment processes as well as with the duration of the holding time of the annealing, the summed results of the metal content for liquid and solid biomass show the highest metal content for processes at 175°C and holding time of 60 min. The determined metal contents of the analysed material should not affect the biological processes of the industry. However, the problem of metal ion contamination due to industrial impacts is a significant and increasingly important issue [31,32].

Unlike organic substances, metal contaminants are not subject to natural decomposition. Heavy metals in the form of ions, on the other hand, are accumulated in the environment, leading to severe environmental contamination [33–36].

5. Conclusions

Investigations of the heavy metal content of steam-explosion-treated GM poplar ash recorded the following observations and conclusions:

1) The highest metallic inhibitor content was usually observed at 175°C. This contamination, although minor, may be of importance when using a closed water circuit due to the accumulation of metallic inhibitors in the effluent. Future studies should investigate whether enzymatic hydrolysis carried out on material contaminated with metallic inhibitors to this extent will have lower efficiency.

2) XRF testing is a simple and rapid method for investigating the content of

metallic elements and the change in their occurrence in the materials under study, which gives the best results for comparative measurements.

3) Dissolution in acid gives a more accurate comparison of values and the most true measurement results due to the homogenisation of the sample and a reduction in measurement uncertainty. The disadvantage of this method is the use of concentrated nitric acid and its neutralisation with ammonia water, accompanied by the release of energy in the form of heat, so it is unsafe. In addition, dissolving the ash leads to a 'dilution' of the metals under test.

4) The process of steam explosion in most of the cases studied results in "washing" of the metals under investigation out of the apparatus. However, it depends on the temperature used, to what extent these metals penetrate into the solid phase that would be subjected to enzymatic hydrolysis after pretreatment. From the point of view of the inhibitory effect of metals in the process, the lowest temperature (160°C) or the highest temperature (215°C) of those proposed should be considered.

5) The liquid phase should also be taken into account during future studies of the heavy metal content of the material after the steam explosion. Although it is the solid phase that is the element of further processing and the metal content in this phase seems to be the most important, determining their content also in the liquid phase can provide a lot of valuable information about the phenomena occurring in the reactor.

 doi:10.20944/preprints202507.2074.v1

Author Contributions: Conceptualization, J.Sz., A.G. and W.J.W; methodology, J.Sz.; software, J.Sz.; validation, J.Sz., A.G. and W.J.W.; formal analysis, J.Sz., A.G. and W.J.W; investigation, J.Sz., A.G.; resources, J.Sz., A.G. and W.J.W.; data curation, J.Sz., A.G. and W.J.W.; writing—original draft preparation, J.Sz., A.G. and W.J.W.; writing—review and editing, J.Sz., A.G. and W.J.W; visualization, J.Sz., A.G. and W.J.W.; supervision, J.Sz., A.G. and W.J.W.; project administration, J.Sz., A.G. and W.J.W.; funding acquisition, J.Sz., A.G. and W.J.W. All authors have read and agreed to the published version of the manuscript.

Funding: Please add: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Acknowledgments: During the preparation of this manuscript, the authors did not use AI for the study results. The authors have reviewed and edited the results and take full responsibility for the content of this publication.

Conflicts of Interest: The Authors declare no conflicts of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

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